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(54) Title: SILICONE VESICLES CONTAINING ACTIVES

(57) Abstract: A process is disclosed for preparing silicone visicle compositions containing a personal care or healthcare active, compositions prepared by the inventive process, and formulated products containing the silicone vesicles.

WO 2005/102248 A2

SILICONE VESICLES CONTAINING ACTIVES

Description

Cross Reference to Related Applications

[0001] This application claims priority to US Patent Application No. 60/563,663 as filed on April 20, 2004, US Patent Application 60/575,915 as filed on June 1, 2004, US Patent Application No. 60/611,258 as filed on September 17, 2004, US Patent Application No. 60/611,229 as filed on September 17, 2004, and US Patent Application No. 60/659,271 as filed on March 7, 2005.

Technical Field

[0002] This invention relates to a process for preparing silicone vesicle compositions containing a personal care or healthcare active, compositions prepared by the inventive process, and formulated personal and healthcare products containing the silicone vesicles.

Background

[0003] Long-standing needs in the field of cosmetic and drug formulation/delivery field are to identify vesicle compositions that form and entrap actives easily, are stable under various chemical and mechanical stresses, and yet are able to deliver the actives in a controlled manner under desired conditions. Vesicles derived from silicone surfactants, and more particularly silicone polyether surfactants, are of interest because of additional inherent benefits that this class of surfactants possesses vs. other types. For example, silicone polyether surfactants often have improved aesthetics in personal care formulations.

Summary

[0004] This invention relates to a process for preparing a vesicle composition comprising;

I) combining,

A) an organopolysiloxane having at least one hydrophilic substituent group,

B) a water miscible volatile solvent,

C) optionally, a silicone or organic oil,

D) a personal care or health care active,

with water to form an aqueous dispersion,

II) mixing the aqueous dispersion to form vesicles,

III) optionally, removing the water miscible volatile solvent from the vesicles.

[0005] The present invention further relates to vesicle compositions prepared by the inventive process, and to personal, household, health care product compositions containing the vesicle compositions.

Detailed Description

[0006] Step I) of the process of the present invention is combining,

A) an organopolysiloxane having at least one hydrophilic substituent group,

B) a water miscible volatile solvent,

C) optionally, a silicone or organic oil,

D) a personal care or health care active,

with water to form an aqueous dispersion.

Components A) – D) are described below.

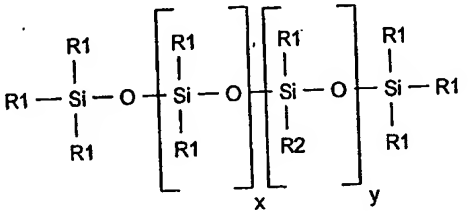
A) Organopolysiloxane component

[0007] Component A) is an organopolysiloxane having at least one hydrophilic substituent group. Organopolysiloxanes are well known in the art and are often designated as comprising any number of "M" siloxy units ($R_3SiO_{0.5}$), "D" siloxy units (R_2SiO), "T" siloxy units ($RSiO_{1.5}$), or "Q" siloxy units (SiO_2) where R is independently any hydrocarbon group. In the present invention, the organopolysiloxane has at least hydrophilic substituent. That is, at least one of the R hydrocarbon groups present in the organopolysiloxane is a hydrophilic group. For purposes of this invention, "hydrophilic group" is the accepted meaning in the art, i.e. designating water loving chemical moieties. Thus, the hydrophilic group can be selected from various cationic, anionic, zwitterionic, polyoxyalkylene, oxoazoline chemical moieties that are commonly used in combination with various hydrophobic chemical moieties to create surfactant structures or molecules having surface-active behavior.

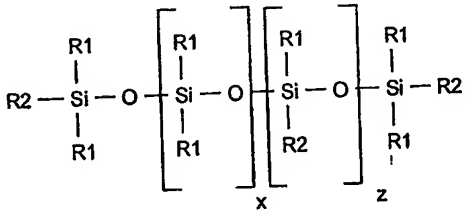
[0008] The amount of the hydrophilic substituent on the organopolysiloxane can vary, depending on the specific chemical component, providing there is at least one hydrophilic group present on the organopolysiloxane. However, the amount of the hydrophilic groups present in the organopolysiloxane can be described by its weight percent, or in particular, the weight

percent of the organopolysiloxane and weight percent of the total hydrophilic groups present in the molecule. Typically, the weight percent of the siloxane units in the organopolysiloxane can vary from 20 to 85, alternatively from 30 to 85, or alternatively from 35 to 80 weight percent, while the remaining weight portion of the organopolysiloxane is the hydrophilic group.

[0009] In one embodiment of the present invention, the organopolysiloxane having at least one hydrophilic substituent group is selected from silicone polyethers. Silicone polyethers (SPEs) generally refer to silicones containing polyether or polyoxyalkylene groups, which could take in many different structural forms. One such form is rake-type SPEs which are derived most commonly from hydrosilylation of SiH functional organosiloxanes with allyloxy-functional polyethers in the presence of a Pt catalyst. In this embodiment, component (A) is a silicone polyether having the structure represented by:



or



[0010] In these structures, R1 represents an alkyl group containing 1-6 carbon atoms such as methyl, ethyl, propyl, butyl, pentyl, and hexyl; R2 represents the group - $(CH_2)_aO(C_2H_4O)_b(C_3H_6O)_cR3$; x has a value of 1-1,000, alternatively 1 - 500, or alternatively 10 - 300; y has a value of 1-500, alternatively 1- 100, or alternatively 2 - 50; z has a value of 1-500, or alternatively 1 - 100; a has a value of 3-6; b has a value of 4-20; c has a value of 0-5; and R3 is hydrogen, a methyl group, or an acyl group such as acetyl. Typically, R1 is methyl; b is 6-12; c is zero; and R3 is hydrogen.

[0011] Preferably, the rake type SPE the silicone polyether has a D/D′ ratio (i.e. x/y ratio) ranging from 5/1 to 50/1, alternatively from 15/1 to 50/1 or alternatively from 20/1 to 50/1.

[0012] In a second embodiment, component (A) is an $(AB)_n$ block silicone polyether (polyorganosiloxane-polyoxyalkylene block copolymer) having the average formula;

[Formula 1] $-[R^1(R_2SiO)_{x'}(R_2SiR^1O)(C_mH_{2m}O)_{y'}]_n-$

where x′ and y′ are greater than 4, m is from 2 to 4 inclusive, n is greater than 2, R is independently a monovalent organic group containing 1 to 20 carbons, R^1 is a divalent hydrocarbon containing 2 to 30 carbons.

[0013] The siloxane block in Formula I is a predominately linear siloxane polymer having the formula $(R_2SiO)_{x'}$, wherein R is independently selected from a monovalent organic group, x′ is a integer greater than 4, alternatively x′ ranges from 20 to 100, or from 30 to 75.

[0014] The organic groups represented by R in the siloxane polymer are free of aliphatic unsaturation. These organic groups may be independently selected from monovalent hydrocarbon and monovalent halogenated hydrocarbon groups free of aliphatic unsaturation. These monovalent groups may have from 1 to 20 carbon atoms, alternatively 1 to 10 carbon atoms, and are exemplified by, but not limited to alkyl groups such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, undecyl, and octadecyl; cycloalkyl such as cyclohexyl; aryl such as phenyl, tolyl, xylyl, benzyl, and 2-phenylethyl; and halogenated hydrocarbon groups such as 3,3,3-trifluoropropyl, 3-chloropropyl, and dichlorophenyl. At least 50 percent, alternatively at least 80%, of the organic groups in the organopolysiloxane may be methyl (denoted as Me). Typically, the siloxane block is a predominately linear polydimethylsiloxane having the formula $(Me_2SiO)_{x'}$, where x′ is as defined above.

[0015] The polyoxyalkylene block of the silicone polyether is represented by the formula $(C_mH_{2m}O)_{y'}$ wherein m is from 2 to 4 inclusive, and y′ is greater than 4, alternatively y′ can range from 5 to 30, or alternatively from 5 to 22. The polyoxyalkylene block typically can comprise oxyethylene units $(C_2H_4O)_{y'}$, oxypropylene units $(C_3H_6O)_{y'}$, oxybutylene units $(C_4H_8O)_{y'}$, or mixtures thereof. Typically, the polyoxyalkylene block comprises oxyethylene units $(C_2H_4O)_{y'}$.

[0016] At least one end of each polyoxyalkylene block in Formula I is linked to a siloxane block by a divalent organic group, designated R^1. This linkage is determined by the reaction employed to prepare the $(AB)_n$ block silicone polyether copolymer. The divalent organic groups of R^1 may be independently selected from divalent hydrocarbons containing 2 to 30 carbons and divalent organofunctional hydrocarbons containing 2 to 30 carbons. Representative, non-limiting examples of such divalent hydrocarbon groups include; ethylene, propylene, butylene, pentylene, hexylene, heptylene, octylene, and the like.

Representative, non-limiting examples of such divalent organofunctional hydrocarbons groups include acrylate and methacrylate. Typically, R^1 is propylene, ($-CH_2CH_2CH_2-$).

[0017] The $(AB)_n$ block silicone polyethers are endblocked. The endblocking unit is also determined by the reaction employed to prepare the $(AB)_n$ block silicone polyether copolymer, which is generally the residual reactive groups of the reactants used. For example, the $(AB)_n$ block silicone polyether copolymers can be prepared by the metal catalyzed hydrosilylation reaction of a diallyl polyether (i.e. an allyl group is present on each molecular terminal end) with a SiH terminated polyorganosiloxane. The resulting $(AB)_n$ block silicone polyether copolymer would have polyoxyalkylene blocks linked to the silicone blocks via a propyleneoxy group ($-CH_2CH_2CH_2O-$), and using a slight molar excess of the allyl polyether would result in an allyl endblock unit ($-CH_2CHCH_2$). Alternative endblock units can result from the addition of other molecules in the reaction employed to prepare the $(AB)_n$ block silicone polyether copolymer that are capable of reacting with the siloxane or polyether block intermediates. For example, the addition of organic compounds having mono-terminated aliphatic unsaturation (such as a mono allyl terminated polyether) will result in the endcapping of the $(AB)_n$ block silicone polyether copolymer with that organic compound. Preferably, the endblocking units of the $(AB)_n$ block silicone polyether is an allyl ether ($CH_2=CHCH_2O-$) or allyl polyether.

[0018] The molecular weights of the $(AB)_n$ block silicone polyether copolymers will be determined by the number of repeating siloxane and polyoxyethylene blocks, as indicated by the subscript n in Formula I. Typically, the value of n is such to provide weight average molecular weights (M_W) to range from 1,500 to 150,000, alternatively, from 10,000 to 100,000.

[0019] The $(AB)_n$ SPEs of the present vesicle compositions have a molar ratio of the total siloxane units to the polyoxyethylene units in the $(AB)_n$ block silicone polyether. This molecular parameter is expressed by the value of $x'/(x'+y')$ in Formula I. The value of $x'/(x'+y')$ can vary from 0.4 to 0.9, or alternatively from 0.55 to 0.9.

[0020] The $(AB)_n$ SPEs useful to prepare the vesicle compositions of the present invention can be prepared by any method known in the art for preparing such block copolymers. Typically however, the $(AB)_n$ SPEs useful in the preparation of the vesicle compositions of the present invention are obtained from a method comprising reacting an SiH terminated organopolysiloxane with a polyoxyethylene having an unsaturated hydrocarbon group at each

molecular terminal, in a hydrosilylation reaction, wherein the mole ratio of the unsaturated hydrocarbon groups to SiH in the reaction is at least 1:1.

B) Water- miscible Volatile Solvent Component

[0021] Component B) is a water-miscible volatile solvent. As used herein "water-miscible" means the solvent forms a dispersion with water at room temperature for at least several hours. "Volatile" means the solvent has a higher vapor pressure than water at various temperatures. As such, when the aqueous dispersion of the organopolysiloxane and solvent are subjected to conditions to remove the solvent, such as heating the dispersion under reduced pressures, the solvent is primarily removed first, allowing all or most of the water to remain in the composition.

[0022] Suitable water-miscible volatile solvents for vesicle dispersion preparation include organic solvents such as alcohols, ethers, glycols, esters, acids, halogenated hydrocarbons, diols. The organic solvents should be miscible with water at the proportion and lower in order to effectively disperse silicones and maintain stable and uniform dispersion overtime. For the purpose of illustration, water-miscible alcohols include method, ethanol, propanol, isopropanol, butanol, and higher hydrocarbon alcohols; ethers include gylcol ethers, methyl-ethyl ether, methyl isobutyl ether (MIBK), etc; glycols include propylene glycols, esters include esters of triglycerol, the esterification products of acid and alcohol; halogenated hydrocarbons include chloroform. Typically water-miscible organic solvents are solvents with relatively low boiling points (<100°C) or high evaporation rate, so they may be removed under vacuum with ease. The most preferred water-miscible organic solvents for this invention are volatile alcohols including methanol, ethanol, isopropanol, and propanol. These alcohols can be removed from aqueous mixtures containing silicone vesicle dispersions via vacuum stripping at ambient temperature.

C) Optional Silicone or Organic Oil Component

[0023] Optional component C) is a silicone or organic oil. The silicone can be any organopolysiloxane having the general formula $R_iSiO_{(4-i)/2}$ in which i has an average value of one to three and R is a monovalent organic group. The organopolysiloxane can be cyclic, linear, branched, and mixtures thereof.

[0024] In one embodiment, component C) is a volatile methyl siloxane (VMS) which includes low molecular weight linear and cyclic volatile methyl siloxanes. Volatile methyl siloxanes conforming to the CTFA definition of cyclomethicones are considered to be within the definition of low molecular weight siloxane.

[0025] Linear VMS have the formula $(CH_3)_3SiO\{(CH_3)_2SiO\}_fSi(CH_3)_3$. The value of f is 0-7. Cyclic VMS have the formula $\{(CH_3)_2SiO\}_g$. The value of g is 3-6. Preferably, these volatile methyl siloxanes have a molecular weight of less than 1,000; a boiling point less than 250 °C; and a viscosity of 0.65 to 5.0 centistoke (mm^2/s), generally not greater than 5.0 centistoke (mm^2/s).

[0026] Representative linear volatile methyl siloxanes are hexamethyldisiloxane (MM) with a boiling point of 100 °C, viscosity of 0.65 mm^2/s, and formula $Me_3SiOSiMe_3$; octamethyltrisiloxane (MDM) with a boiling point of 152 °C, viscosity of 1.04 mm^2/s, and formula $Me_3SiOMe_2SiOSiMe_3$; decamethyltetrasiloxane (MD_2M) with a boiling point of 194 °C, viscosity of 1.53 mm^2/s, and formula $Me_3SiO(Me_2SiO)_2SiMe_3$; dodecamethylpentasiloxane (MD_3M) with a boiling point of 229 °C, viscosity of 2.06 mm^2/s, and formula $Me_3SiO(Me_2SiO)_3SiMe_3$; tetradecamethylhexasiloxane (MD_4M) with a boiling point of 245 °C, viscosity of 2.63 mm^2/s, and formula $Me_3SiO(Me_2SiO)_4SiMe_3$; and hexadecamethylheptasiloxane (MD_5M) with a boiling point of 270 °C, viscosity of 3.24 mm^2/s, and formula $Me_3SiO(Me_2SiO)_5SiMe_3$.

[0027] Representative cyclic volatile methyl siloxanes are hexamethylcyclotrisiloxane (D_3), a solid with a boiling point of 134 °C, a molecular weight of 223, and formula $\{(Me_2)SiO\}_3$; octamethylcyclotetrasiloxane (D_4) with a boiling point of 176 °C, viscosity of 2.3 mm^2/s, a molecular weight of 297, and formula $\{(Me_2)SiO\}_4$; decamethylcyclopentasiloxane (D_5) with a boiling point of 210 °C, viscosity of 3.87 mm^2/s, a molecular weight of 371, and formula $\{(Me_2)SiO\}_5$; and dodecamethylcyclohexasiloxane (D_6) with a boiling point of 245 °C, viscosity of 6.62 mm^2/s, a molecular weight of 445, and formula $\{(Me_2)SiO\}_6$.

[0028] The silicone selected as component C) can be any polydiorganosiloxane fluid, gum, or mixtures thereof. If the polyorganosiloxane has a molecular weight equal to or greater than 1000, it can be blended with the volatile methyl siloxanes described above. The

polydiorganosiloxane gums suitable for the present invention are essentially composed of dimethylsiloxane units with the other units being represented by monomethylsiloxane, trimethylsiloxane, methylvinylsiloxane, methylethylsiloxane, diethylsiloxane, methylphenylsiloxane, diphenylsiloxane, ethylphenylsiloxane, vinylethylsiloxane, phenylvinylsiloxane, 3,3,3-trifluoropropylmethylsiloxane, dimethylphenylsiloxane, methylphenylvinylsiloxane, dimethylethylsiloxane, 3,3,3-trifluoropropyldimethylsiloxane, mono-3,3,3-trifluoropropylsiloxane, aminoalkylsiloxane, monophenylsiloxane, monovinylsiloxane and the like.

[0029] When component C) is an organic oil, it may be selected from any organic oil known in the art suitable for use in the preparation of personal, household, or healthcare formulations. Suitable organic oils include, but are not limited to, natural oils such as coconut oil; hydrocarbons such as mineral oil and hydrogenated polyisobutene; fatty alcohols such as octyldodecanol; esters such as C12 -C15 alkyl benzoate; diesters such as propylene dipelarganate; and triesters, such as glyceryl trioctanoate. The organic oil components can also be mixture of low viscosity and high viscosity oils. Suitable low viscosity oils have a viscosity of 5 to 100 mPa·s at 25°C, and are generally esters having the structure RCO-OR' wherein RCO represents the carboxylic acid radical and wherein OR' is an alcohol residue. Examples of these low viscosity oils include isotridecyl isononanoate, PEG-4 diheptanoate, isostearyl neopentanoate, tridecyl neopentanoate, cetyl octanoate, cetyl palmitate, cetyl ricinoleate, cetyl stearate, cetyl myristate, coco-dicaprylate/caprate, decyl isostearate, isodecyl oleate, isodecyl neopentanoate, isohexyl neopentanoate, octyl palmitate, dioctyl malate, tridecyl octanoate, myristyl myristate, octododecanol, or mixtures of octyldodecanol, acetylated lanolin alcohol, cetyl acetate, isododecanol, polyglyceryl-3-diisostearate, or mixtures thereof. The high viscosity surface oils generally have a viscosity of 200-1,000,000 mPa·s at 25°C, preferably a viscosity of 100,000-250,000 mPa·s. Surface oils include castor oil, lanolin and lanolin derivatives, triisocetyl citrate, sorbitan sesquioleate, C10-18 triglycerides, caprylic/capric/triglycerides, coconut oil, corn oil, cottonseed oil, glyceryl triacetyl hydroxystearate, glyceryl triacetyl ricinoleate, glyceryl trioctanoate, hydrogenated castor oil, linseed oil, mink oil, olive oil, palm oil, illipe butter, rapeseed oil, soybean oil, sunflower seed oil, tallow, tricaprin, trihydroxystearin, triisostearin, trilaurin, trilinolein, trimyristin, triolein, tripalmitin, tristearin, walnut oil, wheat germ oil, cholesterol, or mixtures thereof. Mention may be made, among the optional other non-silicone fatty substances, of

mineral oils, such as liquid paraffin or liquid petroleum, of animal oils, such as perhydrosqualene or arara oil, or alternatively of vegetable oils, such as sweet almond, calophyllum, palm, castor, avocado, jojaba, olive or cereal germ oil. It is also possible to use esters of lanolic acid, of oleic acid, of lauric acid, of stearic acid or of myristic acid, for example; alcohols, such as oleyl alcohol, linoleyl or linolenyl alcohol, isostearyl alcohol or octyldodecanol; or acetylglycerides, octanoates, decanoates or ricinoleates of alcohols or of polyalcohols. It is alternatively possible to use hydrogenated oils which are solid at 25°C, such as hydrogenated castor, palm or coconut oils, or hydrogenated tallow; mono-, di-, tri- or sucroglycerides; lanolins; or fatty esters which are solid at 25°C.

D) Personal or Healthcare Active Component

[0030] Component D) is a personal care or healthcare active. As used herein, a "personal care active" means any compound or mixtures of compounds that are known in the art as additives in the personal care formulations that are typically added for the purpose of treating hair or skin to provide a cosmetic and/or aesthetic benefit. A "healthcare active" means any compound or mixtures of compounds that are known in the art to provide a pharmaceutical or medical benefit. Thus, "healthcare active" include materials consider as an *active ingredient* or *active drug ingredient* as generally used and defined by the United States Department of Health & Human Services Food and Drug Administration, contained in Title 21, Chapter I, of the Code of Federal Regulations, Parts 200-299 and Parts 300-499.

[0031] Thus, *active ingredient* can include any component that is intended to furnish pharmacological activity or other direct effect in the diagnosis, cure, mitigation, treatment, or prevention of disease, or to affect the structure or any function of the body of a human or other animals. The phrase can include those components that may undergo chemical change in the manufacture of drug products and be present in drug products in a modified form intended to furnish the specified activity or effect.

[0032] Some representative examples of *active ingredients* include; drugs, vitamins, minerals; hormones; topical antimicrobial agents such as antibiotic *active ingredients*, antifungal *active ingredients* for the treatment of athlete's foot, jock itch, or ringworm, and acne *active ingredients*; astringent *active ingredients*; deodorant *active ingredients*; wart remover *active ingredients*; corn and callus remover *active ingredients*; pediculicide *active ingredients* for the treatment of head, pubic (crab), and body lice; *active ingredients* for the

control of dandruff, seborrheic dermatitis, or psoriasis; and sunburn prevention and treatment agents.

[0033] Useful *active ingredients* for use in processes according to the invention include both fat or oil-soluble vitamins as well as water-soluble vitamins. Oil-soluble vitamins useful herein include, but are not limited to, Vitamin A_1, RETINOL, C_2-C_{18} esters of RETINOL, vitamin E, TOCOPHEROL, esters of vitamin E, and mixtures thereof. RETINOL includes trans-RETINOL, 1, 3-cis-RETINOL, 11-cis-RETINOL, 9-cis-RETINOL, and 3,4-didehydro-RETINOL.

[0034] RETINOL, it should be noted, is an International Nomenclature Cosmetic Ingredient Name (INCI) designated by The Cosmetic, Toiletry, and Fragrance Association (CTFA), Washington DC, for vitamin A. Other suitable vitamins and the INCI names for the vitamins considered included herein are RETINYL ACETATE, RETINYL PALMITATE, RETINYL PROPIONATE, α-TOCOPHEROL, TOCOPHERSOLAN, TOCOPHERYL ACETATE, TOCOPHERYL LINOLEATE, TOCOPHERYL NICOTINATE, and TOCOPHERYL SUCCINATE.

[0035] Water-soluble vitamins useful herein include, but are not limited to, Vitamin C, Vitamin B_1, Vitamin B_2, Vitamin B_6, Vitamin B_{12}, niacin, folic acid, biotin, and pantothenic acid. Other suitable water-soluble vitamins and the INCI names for the vitamins considered included herein are ASCORBYL DIPALMITATE, ASCORBYL METHYLSILANOL PECTINATE, ASCORBYL PALMITATE, and ASCORBYL STEARATE.

[0036] Some examples of commercially available products suitable for use herein are Vitamin A Acetate and Vitamin C, both products of Fluka Chemie AG, Buchs, Switzerland; COVI-OX T-50, a vitamin E product of Henkel Corporation, La Grange, Illinois; COVI-OX T-70, another vitamin E product of Henkel Corporation, La Grange, Illinois; and vitamin E Acetate, a product of Roche Vitamins & Fine Chemicals, Nutley, New Jersey.

[0037] The *active ingredient* used in processes according to the invention can be a water-soluble or an oil-soluble *active drug ingredient*. Representative examples of some suitable water-soluble *active drug ingredients* which can be used are hydrocortisone, ketoprofen, timolol, pilocarpine, adriamycin, mitomycin C, morphine, hydromorphone, diltiazem, theophylline, doxorubicin, daunorubicin, heparin, penicillin G, carbenicillin, cephalothin,

cefoxitin, cefotaxime, 5-fluorouracil, cytarabine, 6-azauridine, 6-thioguanine, vinblastine, vincristine, bleomycin sulfate, aurothioglucose, suramin, and mebendazole.

[0038] Representative examples of some suitable oil-soluble *active drug ingredients* which can be used are clonidine, scopolamine, propranolol, phenylpropanolamine hydrochloride, ouabain, atropine, haloperidol, isosorbide, nitroglycerin, ibuprofen, ubiquinones, indomethacin, prostaglandins, naproxen, salbutamol, guanabenz, labetalol, pheniramine, metrifonate, and steroids.

[0039] Considered to be included herein as *active drug ingredients* for purposes of the present invention are antiacne agents such as benzoyl peroxide and tretinoin; antibacterial agents such as chlorohexadiene gluconate; antifungal agents such as miconazole nitrate; anti-inflammatory agents; corticosteroidal drugs; non-steroidal anti-inflammatory agents such as diclofenac; antipsoriasis agents such as clobetasol propionate; anesthetic agents such as lidocaine; antipruritic agents; antidermatitis agents; and agents generally considered barrier films.

[0040] The active component D) of the present invention can be a protein, such as an enzyme. Enzymes include, but are not limited to, commercially available types, improved types, recombinant types, wild types, variants not found in nature, and mixtures thereof. For example, suitable enzymes include hydrolases, cutinases, oxidases, transferases, reductases, hemicellulases, esterases, isomerases, pectinases, lactases, peroxidases, laccases, catalases, and mixtures thereof. Hydrolases include, but are not limited to, proteases (bacterial, fungal, acid, neutral or alkaline), amylases (alpha or beta), lipases, mannanases, cellulases, collagenases and mixtures thereof.

[0041] Component D) may also be a sunscreen agent. The sunscreen agent can be selected from any sunscreen agent known in the art to protect skin from the harmful effects of exposure to sunlight. The sunscreen can be an organic compound, an inorganic compound, or mixtures thereof. Thus, representative non limiting examples that can be used as the sunscreen agent include; Aminobenzoic Acid, Cinoxate, Diethanolamine Methoxycinnamate, Digalloyl Trioleate, Dioxybenzone, Ethyl 4-[bis(Hydroxypropyl)] Aminobenzoate, Glyceryl Aminobenzoate, Homosalate, Lawsone with Dihydroxyacetone, Menthyl Anthranilate, Octocrylene, Octyl Methoxycinnamate, Octyl Salicylate, Oxybenzone, Padimate O, Phenylbenzimidazole Sulfonic Acid, Red Petrolatum, Sulisobenzone, Titanium Dioxide, and Trolamine Salicylate.

[0042] The organic sunscreen compound is typically chosen from an organic compound that absorbs ultraviolet (UV) light. Some examples of UV light absorbing compounds are Acetaminosalol, Allatoin PABA, Benzalphthalide, Benzophenone, Benzophenone 1-12, 3-Benzylidene Camphor, Benzylidenecamphor Hydrolyzed Collagen Sulfonamide, Benzylidene Camphor Sulfonic Acid, Benzyl Salicylate, Bornelone, Bumetriozole, Butyl Methoxydibenzoylmethane, Butyl PABA, Ceria/Silica, Ceria/Silica Talc, Cinoxate, DEA-Methoxycinnamate, Dibenzoxazol Naphthalene, Di-t-Butyl Hydroxybenzylidene Camphor, Digalloyl Trioleate, Diisopropyl Methyl Cinnamate, Dimethyl PABA Ethyl Cetearyldimonium Tosylate, Dioctyl Butamido Triazone, Diphenyl Carbomethoxy Acetoxy Naphthopyran, Disodium Bisethylphenyl Tiamminotriazine Stilbenedisulfonate, Disodium Distyrylbiphenyl Triaminotriazine Stilbenedisulfonate, Disodium Distyrylbiphenyl Disulfonate, Drometrizole, Drometrizole Trisiloxane, Ethyl Dihydroxypropyl PABA, Ethyl Diisopropylcinnamate, Ethyl Methoxycinnamate, Ethyl PABA, Ethyl Urocanate, Etrocrylene Ferulic Acid, Glyceryl Octanoate Dimethoxycinnamate, Glyceryl PABA, Glycol Salicylate, Homosalate, Isoamyl p-Methoxycinnamate, Isopropylbenzyl Salicylate, Isopropyl Dibenzolylmethane, Isopropyl Methoxycinnamate, Menthyl Anthranilate, Menthyl Salicylate, 4-Methylbenzylidene, Camphor, Octocrylene, Octrizole, Octyl Dimethyl PABA, Octyl Methoxycinnamate, Octyl Salicylate, Octyl Triazone, PABA, PEG-25 PABA, Pentyl Dimethyl PABA, Phenylbenzimidazole Sulfonic Acid, Polyacrylamidomethyl Benzylidene Camphor, Potassium Methoxycinnamate, Potassium Phenylbenzimidazole Sulfonate, Red Petrolatum, Sodium Phenylbenzimidazole Sulfonate, Sodium Urocanate, TEA-Phenylbenzimidazole Sulfonate, TEA-Salicylate, Terephthalylidene Dicamphor Sulfonic Acid, Titanium Dioxide, TriPABA Panthenol, Urocanic Acid, and VA/Crotonates/Methacryloxybenzophenone-1 Copolymer.

[0043] Alternatively, the sunscreen agent is a cinnamate based organic compound, or alternatively, the sunscreen agent is octyl methoxycinnamate, such as Uvinul® MC 80 an ester of para-methoxycinnamic acid and 2-ethylhexanol.

[0044] Component D) may also be a fragrance or perfume. The perfume can be any perfume or fragrance active ingredient commonly used in the perfume industry. These compositions typically belong to a variety of chemical classes, as varied as alcohols, aldehydes, ketones, esters, ethers, acetates, nitrites, terpenic hydrocarbons, heterocyclic nitrogen or sulfur containing compounds, as well as essential oils of natural or synthetic

origin. Many of these perfume ingredients are described in detail in standard textbook references such as *Perfume and Flavour Chemicals*, 1969, S. Arctander, Montclair, New Jersey.

[0045] Fragrances may be exemplified by, but not limited to, perfume ketones and perfume aldehydes. Illustrative of the perfume ketones are buccoxime; iso jasmone; methyl beta naphthyl ketone; musk indanone; tonalid/musk plus; Alpha-Damascone, Beta-Damascone, Delta-Damascone, Iso-Damascone, Damascenone, Damarose, Methyl-Dihydrojasmonate, Menthone, Carvone, Camphor, Fenchone, Alpha-lonone, Beta-lonone, Gamma-Methyl so-called lonone, Fleuramone, Dihydrojasmone, Cis-Jasmone, Iso-E-Super, Methyl-Cedrenyl-ketone or Methyl- Cedrylone, Acetophenone, Methyl-Acetophenone, Para-Methoxy-Acetophenone, Methyl-Beta-Naphtyl-Ketone, Benzyl-Acetone, Benzophenone, Para-Hydroxy-Phenyl-Butanone, Celery Ketone or Livescone, 6-Isopropyldecahydro-2-naphtone, Dimethyl-Octenone, Freskomenthe, 4-(1-Ethoxyvinyl)-3,3,5,5,-tetramethyl-Cyclohexanone, Methyl-Heptenone, 2-(2-(4-Methyl-3-cyclohexen-1-yl)propyl)-cyclopentanone, 1-(p-Menthen-6(2)-yl)-1-propanone, 4-(4-Hydroxy-3-methoxyphenyl)-2-butanone, 2-Acetyl-3,3-Dimethyl-Norbomane, 6,7-Dihydro-1,1,2,3,3-Pentamethyl-4(5H)-Indanone, 4-Damascol, Dulcinyl or Cassione, Gelsone, Hexalon, Isocyclemone E, Methyl Cyclocitrone, Methyl-Lavender-Ketone, Orivon, Para-tertiary-Butyl-Cyclohexanone, Verdone, Delphone, Muscone, Neobutenone, Plicatone, Veloutone, 2,4,4,7-Tetramethyl-oct-6-en-3-one, and Tetrameran.

[0046] More preferably, the perfume ketones are selected for its odor character from Alpha Damascone, Delta Damascone, Iso Damascone, Carvone, Gamma-Methyl-lonone, Iso-E-Super, 2,4,4,7-Tetramethyl-oct-6-en-3-one, Benzyl Acetone, Beta Damascone, Damascenone, methyl dihydrojasmonate, methyl cedrylone, and mixtures thereof.

[0047] Preferably, the perfume aldehyde is selected for its odor character from adoxal; anisic aldehyde; cymal; ethyl vanillin; florhydral; helional; heliotropin; hydroxycitronellal; koavone; lauric aldehyde; lyral; methyl nonyl acetaldehyde; P. T. bucinal; phenyl acetaldehyde; undecylenic aldehyde; vanillin; 2,6,10-trimethyl-9-undecenal, 3-dodecen-1-al, alpha-n-amyl cinnamic aldehyde, 4-methoxybenzaldehyde, benzaldehyde, 3-(4-tert butylphenyl)-propanal, 2-methyl-3-(para-methoxyphenyl propanal, 2-methyl-4-(2,6,6-trimethyl-2(1)-cyclohexen-1-yl) butanal, 3-phenyl-2-propenal, cis-/trans-3,7-dimethyl-2,6-octadien-1-al, 3,7-dimethyl-6-octen-1-al, [(3,7-dimethyl-6-octenyl)oxy] acetaldehyde, 4-

isopropylbenzyaldehyde, 1,2,3,4,5,6,7,8-octahydro-8,8-dimethyl-2-naphthaldehyde , 2,4-dimethyl-3-cyclohexen-1-carboxaldehyde, 2-methyl-3-(isopropylphenyl)propanal, 1-decanal; decyl aldehyde, 2,6-dimethyl-5-heptenal, 4-(tricyclo[5.2.1.0(2,6)]-decylidene-8)-butanal, octahydro-4,7-methano-1H- indenecarboxaldehyde, 3-ethoxy-4-hydroxy benzaldehyde, para-ethyl-alpha, alpha-dimethyl hydrocinnamaldehyde, alpha-methyl-3,4-(methylenedioxy)-hydrocinnamaldehyde, 3,4-methylenedioxybenzaldehyde, alpha-n-hexyl cinnamic aldehyde, m-cymene-7-carboxaldehyde, alpha-methyl phenyl acetaldehyde, 7-hydroxy-3,7-dimethyl octanal, Undecenal, 2,4,6-trimethyl-3-cyclohexene-1-carboxaldehyde, 4-(3)(4-methyl-3-pentenyl)-3-cyclohexen-carboxaldehyde, 1-dodecanal, 2,4-dimethyl cyclohexene-3-carboxaldehyde, 4-(4-hydroxy-4-methyl pentyl)-3-cylohexene-1-carboxaldehyde, 7-methoxy-3,7-dimethyloctan-1-al, 2-methyl undecanal, 2-methyl decanal, 1-nonanal, 1-octanal, 2,6,10-trimethyl-5,9-undecadienal, 2-methyl-3-(4-tertbutyl)propanal, dihydrocinnamic aldehyde, 1-methyl-4-(4-methyl-3-pentenyl)-3-cyclohexene-1-carbox aldehyde, 5 or 6 methoxy1 0 hexahydro-4,7-methanoindan-1 or 2- carboxaldehyde, 3,7-dimethyloctan-1-al, 1 -undecanal, 10-undecen-1-al, 4-hydroxy-3-methoxy benzaldehyde, 1-methyl-3-(4-methylpentyl)-3-cyclhexenecarboxaldehyde, 7-hydroxy-3,7-dimethyl-octanal, trans-4-decenal, 2,6-nonadienal, paratolylacetaldehyde; 4-methylphenylacetaldehyde, 2-methyl-4-(2,6,6-trimethyl-1-cyclohexen-1-yl)-2-butena l, ortho-methoxycinnamic aldehyde, 3,5,6-trimethyl-3-cyclohexene carboxaldehyde, 3,7-dimethyl-2-methylene-6-octenal, phenoxyacetaldehyde, 5,9-dimethyl-4,8-decadienal, peony aldehyde (6,10-dimethyl-3-oxa-5,9-undecadien-1-al), hexahydro-4,7-methanoindan-1-carboxaldehyde, 2-methyl octanal, alpha-methyl-4-(1-methyl ethyl) benzene acetaldehyde, 6,6-dimethyl-2-norpinene-2-propionaldehyde, para methyl phenoxy acetaldehyde, 2-methyl-3-phenyl-2-propen-1-al, 3,5,5-trimethyl hexanal, Hexahydro-8,8-dimethyl-2-naphthaldehyde, 3-propyl-bicyclo[2.2.1]-hept-5-ene-2-carbaldehyde, 9-decenal, 3-methyl-5-phenyl-1-pentanal, methylnonyl acetaldehyde, hexanal, trans-2-hexenal, 1-p-menthene-q-carboxaldehyde and mixtures thereof.

[0048] More preferred aldehydes are selected for their odor character from 1-decanal, benzaldehyde, florhydral, 2,4-dimethyl-3-cyclohexen-1-carboxaldehyde; cis/trans-3,7-dimethyl-2,6-octadien-1-al; heliotropin; 2,4,6-trimethyl-3-cyclohexene-1-carboxaldehyde; 2,6-nonadienal; alpha-n-amyl cinnamic aldehyde, alpha-n-hexyl cinnamic aldehyde, P.T.

Bucinal, lyral, cymal, methyl nonyl acetaldehyde, hexanal, trans-2-hexenal, and mixture thereof.

[0049] In the above list of perfume ingredients, some are commercial names conventionally known to one skilled in the art, and also includes isomers. Such isomers are also suitable for use in the present invention.

[0050] The amount of components A), B), C), and D) can vary in the process, but typically range as follows;

A) 2 to 50 wt%, alternatively 2 to 25 wt %, or alternatively 2 to 15 wt%,

B) 1 to 50 wt%, alternatively 2 to 25 wt %, or alternatively 2 to 15 wt%,

C) 0 to 50 wt %, alternatively 1 to 20 wt %, or alternatively 2 to 10 wt%,

D) 0.05 to 20 wt%, alternatively 0.1 to 10 wt %, or alternatively 0.1 to 5 wt%,

and sufficient amount of water to provide the sum of the wt% of A), B), and C) and water content to equal 100%.

[0051] The order of combining components A), B), C), and D) with water is not critical, but typically A), B), C), and D) are first combined and then added with water to form an aqueous dispersions of components A) – D).

[0052] Step II in the process of the present invention is mixing the aqueous dispersion formed in Step I to form vesicles. There are no special requirements or conditions needed to effect the mixing and formation of vesicles. Mixing techniques can be simple stirring, homogenizing, sonalating, and other mixing techniques known in the art to effect the formation of vesicles in aqueous dispersions. The mixing can be conducted in a batch, semi-continuous, or continuous process.

[0053] The formation of vesicles can be confirmed by techniques common in the state of the art. Typically, vesicles have a lamellar phase structure which exhibit birefringence when examined with a cross polarizing microscope. Alternatively, the formation of vesicles can be demonstrated by Cyro-Transmission Electron Microscopy (Cryo-TEM) techniques. Particle size measurements can also be used to indicate that the organopolysiloxanes are sufficiently dispersed in aqueous medium typical of vesicle sizes. For example, average particle sizes of less than 0.500 μm (micrometers), are typical for dispersed vesicles. Vesicles having an average particle size of less than 0.200 μm, or 0.100 μm are possible with the teachings of the present invention.

[0054] Step III in the process of the present invention is optional, and involves removing the water miscible volatile solvent, component B). Typically, the water miscible volatile solvent is removed by known techniques in the art, such as subjecting the vesicle composition to reduced pressures, while optionally heating the composition. Devices illustrative of such techniques include rotary evaporators and thin film strippers.

[0055] The present invention further encompasses the vesicle compositions prepared by the process described herein.

[0056] The vesicle compositions prepared according to the invention can be used in various over-the-counter (OTC) personal care compositions, health care compositions, and household care compositions, but especially in the personal care arena. The vesicle compositions prepared according to the present invention can be combined with a variety of personal, household, or healthcare ingredients in a formulated product composition. A listing of possible personal, household, or health care ingredients is taught in WO 03/101412, which is incorporated herein by reference. Thus, they can be used in antiperspirants, deodorants, skin creams, skin care lotions, moisturizers, facial treatments such as acne or wrinkle removers, personal and facial cleansers, bath oils, perfumes, colognes, sachets, sunscreens, pre-shave and after-shave lotions, liquid soaps, shaving soaps, shaving lathers, hair shampoos, hair conditioners, hair sprays, mousses, permanents, depilatories, hair cuticle coats, make-ups, color cosmetics, foundations, blushes, lipsticks, lip balms, eyeliners, mascaras, oil removers, color cosmetic removers, nail polishes, and powders.

[0057] The vesicle compositions can be combined with a powder to provide a formulation base for a variety of cosmetic products. A powder is defined herein as a dry particulate matter having a particle size of 0.02-50 microns. The particulate matter may be colored or non-colored (for example white). Suitable powders include bismuth oxychloride, titanated mica, fumed silica, spherical silica beads, polymethylmethacrylate beads, micronized teflon, boron nitride, acrylate polymers, aluminum silicate, aluminum starch octenylsuccinate, bentonite, calcium silicate, cellulose, chalk, corn starch, diatomaceous earth, fuller's earth, glyceryl starch, hectorite, hydrated silica, kaolin, magnesium aluminum silicate, magnesium carbonate, magnesium hydroxide, magnesium oxide, magnesium silicate, magnesium trisilicate, maltodextrin, montmorillonite, microcrystalline cellulose, rice starch, silica, talc, mica, titanium dioxide, zinc laurate, zinc myristate, zinc neodecanoate, zinc rosinate, zinc stearate, polyethylene, alumina, attapulgite, calcium carbonate, calcium silicate, dextran,

kaolin, nylon, silica silylate, silk powder, serecite, soy flour, tin oxide, titanium hydroxide, trimagnesium phosphate, walnut shell powder, or mixtures thereof. The above mentioned powders may be surface treated with lecithin, amino acids, mineral oil, silicone oil, or various other agents either alone or in combination, which coat the powder surface and render the particles hydrophobic in nature.

[0058] The powder may also comprise various organic and inorganic pigments. The organic pigments are generally various aromatic types including azo, indigoid, triphenylmethane, anthraquinone, and xanthine dyes which are designated as D&C and FD&C blues, browns, greens, oranges, reds, yellows, etc. Inorganic pigments generally consist of insoluble metallic salts of certified color additives, referred to as the Lakes or iron oxides. A pulverulent coloring agent, such as carbon black, chromium or iron oxides, ultramarines, manganese pyrophosphate, iron blue, and titanium dioxide, pearlescent agents, generally used as a mixture with colored pigments, or some organic dyes, generally used as a mixture with colored pigments and commonly used in the cosmetics industry, can be added to the composition. Pulverulent inorganic or organic fillers can also be added. These pulverulent fillers can be chosen from talc, micas, kaolin, zinc or titanium oxides, calcium or magnesium carbonates, silica, spherical titanium dioxide, glass or ceramic beads, metal soaps derived from carboxylic acids having 8-22 carbon atoms, non-expanded synthetic polymer powders, expanded powders and powders from natural organic compounds, such as cereal starches, which may or may not be crosslinked. Mention may be made in particular of talc, mica, silica, kaolin, nylon powders (in particular ORGASOL), polyethylene powders, Teflon, starch, boron nitride, copolymer microspheres such as EXPANCEL (Nobel Industrie), POLYTRAP, and silicone resin microbeads (TOSPEARL from Toshiba, for example).

Examples

[0059] These examples are intended to illustrate the invention to one of ordinary skill in the art and should not be interpreted as limiting the scope of the invention set forth in the claims.

Example 1: (reference) preparation of vesicle compositions

[0060] A vesicle composition (labeled as 1A) was prepared from a rake silicone polyether, having a nominal structure of $MD_{94}D^{(EO12)}{}_6M$, where M represents $(CH_3)_3SiO_{1/2}$ siloxy units, D represent $(CH_3)_2SiO$ siloxy units, $(CH_3)R^{EO12}SiO$ siloxy units where R^{EO12} represents the polyethylene oxide group having the average formula, $-CH_2CH_2CH_2O(CH_2CH_2O)_{12}H$. A vesicle composition (labeled as 1B) was also prepared from a $(AB)_n$ SPE block copolymer of $M'D_{50}M'$ siloxane (where M represents $(CH_3)_2HSiO_{1/2}$ siloxy units, D represent $(CH_3)_2SiO$ siloxy units) and Polyglycol AA1200 polyether (α,ω–diallyl polyethylene oxide having an average molecular weight (M_w) of 1200). Both vesicle compositions were processed to entrap vitamin A palmitate as a representative example of an active material. These vesicle compositions were made via processing in ethanol / water media. The composition and initial vesicle properties are summarized in Table 1.

Table 1

Vesicle Example ID	**1A**	**1B**
SPE type	**rake SPE**	**$(AB)_n$ SPE**
Wt. % silicone polyether	**20.90**	**21.27**
Wt. % vitamin A palmitate	**4.50**	**4.59**
Wt. % carrier fluid + additive	1.81	1.84
Wt. % Water	**72.80**	**72.30**
Wt % Loading	17.71	17.77
Initial property		
Vesicle dispersion appearance	Pale yellow fluid	Very pale yellow fluid
Average size Mv, μm	**0.0735**	**0.255**
D(v, 0.5), μm	0.0592	0.2288
D(v, 0.9), μm	0.1361	0.447

Example 2: Moisturizing gel

Part A	
1. Water	q.s.
2. DMDM Hydantoin (Nipaguard DMDMH, Clariant GmbH)	0.30%
3. Acrylates/ C10-30 Alkyl acrylate cross polymer	
4. (Carbopol ETD 2020, Noveon)	1%
5. Triethanolamine (30%)	q.s.
Part B	
6. Silicone Vesicles of Example 1	q.s.

Example 3- Moisturizing gel

Part A	
1. Water	q.s.
2. DMDM Hydantoin (Nipaguard DMDMH, Clariant GmbH) 0.30%	
3. Polyacrylamide, C13-14 Isoparafin, Laureth-7 (Sepigel 305, Seppic)	1%
Part B	
4. Silicone Vesicles of Example 1	q.s.

Procedure:

- Prepare gel in Part A
- Add part B
- Mix to homogeneous

Example 4: O/W Body lotion

An oil-in-water body lotion is prepared following the following procedure. Silicone vesicles can also be formulated into other oil-in-water type cosmetic formulations.

Part A	
1. Cetearyl Alcohol (Lanette O, Cognis)	3%
2. Diisopropyl Adipate (Crodamol DA, Croda)	5%
3. Dimethicone (Dow Corning® 200 fluid, 100 cSt)	0.5%
4. Potassium cetyl phosphate (Amphisol K, Roche Vitamins)	1.5%
5. Buthylated hydroxytoluene (BHT)	0.05%
6. Tetrasodium EDTA	0.1%
7. Phenoxyethanol, methyl paraben, ethyl paraben, propyl paraben, butylparaben (Phenonip, Clariant)	0.6%
Part B	
8. Water	q.s.
9. Carbomer (1%) (Carbopol 980, Noveon)	30%
10. Potassium hydroxyde (10%)	1.5%

Part C

11. Silicone Vesicles of Example 1 q.s.

Procedure

- Heat Part A to 85°C while stirring
- When homogeneous, add Part B at 40°C
- Let cool down to room temperature and compensate for water loss
- Add part C to AB
- Mix to homogeneous

Example 5: W/O Radiant Beauty Formulation

Silicone vesicles are formulated into water-in-oil radiant beauty formulation following the procedures listed below. Other water-in-oil type personal care formulations can also be prepared following these similar steps.

Part A

1. Ethylhexyl Methoxycinnamate (Parsol MCX, Roche Vitamins) 3%
2. Butyl Methoxydibenzoylmethane (Parsol 1789, Roche Vitamins) 1.5%
3. Glyceryl Stearate, PEG 100 stearate (Arlacel 165, Uniqema) 4%
4. Butyrospermum Parkii, Shea butter (Cetiol SB 45, Cognis) 1%
5. Stearyl dimethicone (Dow Corning® 2503 Cosmetic wax) 3%
6. Cetyl alcohol 1%
7. Simmondsia Chinensis (Jojoba) Seed Oil 4%
8. Lanolin oil (Fluilan, Croda) 3%
9. Cyclomethicone (Dow Corning® 245 Fluid) 8%
10. Phenoxyethanol, methyl paraben, ethyl paraben, propyl paraben, butylparaben (Phenonip, Clariant) 0.5%
11. Cyclomethicone (and) Dimethicone Crosspolymer (Dow Corning® 9045 Silicone Elastomer Blend) 5%

Part B

12. Glycerin 2%
13. Water q.s.

Part C

14. Polyacrylamide, C13-14 Isoparafin, Laureth-7 (Sepigel 305, Seppic) 4%

Part D

15. Silicone Vesicles of Example 1 q.s.

Procedure

- Melt ingredients 1 and 2 at 60°C
- Add ingredients 3, 4, 5, 6 in order at 60°C, ensuring that each ingredient is melted before incorporating the next
- Add 7, 8, 9 and 10
- Add ingredient 11 to form Part A
- Add Part B to Part A at 1500 rpm
- Let cool down to room temperature
- Add Part C to AB in "one shot" while stirring at maximum speed
- Cease agitation immediately once viscosity increases
- Add Part D to ABC
- Mix to homogeneous

Example 6: Mild Foundation

Color cosmetic products are prepared from silicone vesicles and other cosmetic pigments and colorants. Illustrated in this example is a mild foundation containing silicone vesicles, following the procedure shown bellow.

Part A

1. Polyglyceryl-4 Caprate (and) Sucrose Stearate (and) Sucrose Distearate (and) PEG-8 (and) Ammonium Polyacrylate (and) Mica (and) Tocopheryl Acetate (and) Macadamia Ternifolia Seed Extract (Covacream, Sensient Cosmetic Technology - LCW) 5.00%

Part B

2. Hydrogenated Polyisobutene (Squatol S, Sensient Cosmetic Technology - LCW) 10.00%
3. Cyclomethicone (Dow Corning® 245 Fluid) 5.00%

Part C

4. Glycerin 2.50%
5. DMDM Hydantoin (Nipaguard DMDMH, Clariant GmbH) 0.30%
6. Sodium polyacrylate (Covacryl J22, Sensient Cosmetic Technology - LCW) 0.30%
7. Water q.s.

Part D

8. Red iron oxide (AQ 70401, Sensient Cosmetic Technology - LCW) 0.50%
9. Yellow iron oxide (AQ 70402, Sensient Cosmetic Technology - LCW) 0.90%
10. Black iron oxide (AQ 70403, Sensient Cosmetic Technology - LCW) 0.10%
11. Titanium Dioxide (AQ 70409, Sensient Cosmetic Technology - LCW) 8.50%

Part E

12. Silicone Vesicles of Example 1 q.s.

Procedure

- Mix ingredients of Part B together
- Add Part B to Part A under stirring - Part AB will look heterogeneous
- Mix Part C ingredients together
- Add Part C to Part AB and stir until homogeneous
- Mix Part D ingredients together
- Add Part D to the batch, stir under high shear during 30 minutes
- When this is done, add Part E
- Mix to homogeneous

Example 7: Emulsion lipstick

This example illustrates how silicone vesicles can be used in formulating color cosmetic products including lipstick.

Part A

1. Lauryl PEG/PPG-18/18 Methicone (Dow Corning 5200 Formulation Aid) 3.70%
2. Phenyl Trimethicone (Dow Corning 556 Cosmetic Grade Fluid) 1.00%
3. Hexyl Laurate (Cetiol A, Cognis Corporation, Care Chemicals) 3.50%
4. Disteardimonium Hectorite (Bentone 38, Elementis Specialties) 0.30%
5. Isononyl Isononanoate (and) Polybutene (and) Pentaerythrityl Tetraisostearate (and) Isostearyl Alcohol (Covaclear, Sensient Cosmetic Technology - LCW) 2.40%
6. Iron Oxides (Unipure Red LC 381 AS-EM, Sensient Cosmetic Technology - LCW) 3.00%

Part B

7. Pure water q.s.
8. Algae Extract (and) Sorbitol (Fucosorb, Sensient Cosmetic Technology - LCW) 0.60%
9. Propylene glycol 1.20%

Part C

10. Ozokerite (and) Copernicia Cerifera (Carnauba) Wax (and) Euphorbia Cerifera (Candelilla) Wax (and) Paraffin (and) Butyl Stearate (and) Isopropyl Palmitate (and) Mineral Oil (and) Ethylene/VA Copolymer (Covalip 94, Sensient Cosmetic Technology - LCW) 20.00%
11. Ethylhexyl Hydroxystearate (and) Triethylhexyl Trimellitate (and) C30-45 Olefin (Clearwax, Sensient Cosmetic Technology - LCW) 1.50%

12. Octyldodecanol (Eutanol G, Cognis Corporation, Care Chemicals) 18.50%
13. Isononyl Isononanoate (and) Polybutene (and) Pentaerythrityl Tetraisostearate (and) Isostearyl Alcohol (Covaclear, Sensient Cosmetic Technology - LCW) 18.00%

Part D

14. Red iron oxide (AS 70421, Sensient Cosmetic Technology - LCW) 5.00%
15. Yellow iron oxide (AS 70422, Sensient Cosmetic Technology - LCW) 0.30%
16. Black iron oxide (AS 70423, Sensient Cosmetic Technology - LCW) 0.30%
17. Titanium Dioxide (Unipure White LC 981 AS, Sensient Cosmetic Technology - LCW) 6.30%

Part E q.s.

18. Silicone Vesicles of Example 1

Procedure

a. Disperse well the Iron Oxide (Unipure Red LC381) in the rest of phase A while stirring
b. Prepare phase B and pour phase B into phase A under stirring
c. Prepare phase D
d. Heat phase C to 60°C until the waxes are melted
e. Add phase D to phase C
f. Heat AB to 50°C
g. Add AB in CD (normally after complete removal of the bubbles) at 50°C
h. Then let cool down to 40-45°C and add part E

Mix to homogeneous

CLAIMS

1. A process for preparing a vesicle composition comprising;

I) combining,

A) an organopolysiloxane having at least one hydrophilic substituent group,

B) a water miscible volatile solvent,

C) optionally, a silicone or organic oil,

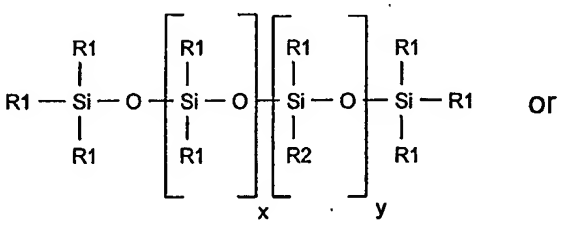
D) a personal care or health care active,

with water to form an aqueous dispersion,

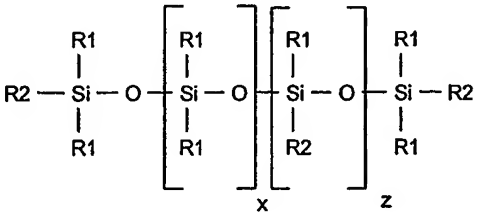
II) mixing the aqueous dispersion to form vesicles,

III) optionally, removing the water miscible volatile solvent from the vesicles.

2. The process of claim 1 wherein the organopolysiloxane is a silicone polyether having the formula:



or



where R1 represents an alkyl group containing 1-6 carbon atoms;

R2 represents the group $-(CH_2)_aO(C_2H_4O)_b(C_3H_6O)_cR3$;

x is 1-1,000; y is 1-500; z is 1-500; a is 3-6; b is 4-20; c is 0-5;

and R3 is hydrogen, a methyl group, or an acyl group.

3. The process of claim 1 wherein the organopolysiloxane is a $(AB)_n$ block silicone polyether having the formula;

$$-[R^1(R_2SiO)_{x'}(R_2SiR^1O)(C_mH_{2m}O)_{y'}]_n-$$

where x′ and y′ are greater than 4, m is from 2 to 4 inclusive, n is greater than 2, R is independently a monovalent organic group containing 1 to 20 carbons, R^1 is a divalent hydrocarbon containing 2 to 30 carbons.

4. The process of claim 1wherein the water miscible volatile solvent is an alcohol.

5. The process of claim 4 wherein the alcohol is ethanol or isopropanol.

6. The process of claim 1 wherein component C) is present and is a volatile methyl siloxane.

7. The process of claim 1 wherein the personal care active is a vitamin.

8. The process of claim 7 wherein the vitamin is Vitamin A palmitate.

9. The process of claim 1 wherein the personal care active is a sunscreen agent.

10. The process of claim 1 wherein the personal care active is a fragrance or perfume.

11. The process of claim 1 wherein the healthcare active is a pharmaceutical drug.

12. A vesicle composition prepared according to the process of any one of claims 1-11.

13. A personal care product comprising the vesicle composition of claim 12.

14. The personal care product of claim 13 wherein the personal care product is selected from an antiperspirant, deodorant, skin cream, skin care lotion, moisturizer, facial treatment, wrinkle remover, facial cleansers, bath oils, sunscreens, pre-shave, after-shave lotions, liquid soap, shaving soap, shaving lather, hair shampoo, hair conditioner, hair spray, mousse, permanent, hair cuticle coat, make-up, color cosmetic, foundation, blush, lipstick, lip balm, eyeliner, mascara, nail polishes, and powders.

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Declarations under Rule 4.17:

— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii)) for all designations*

— *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii)) for the following designations AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW, ARIPO patent (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG)*

— *of inventorship (Rule 4.17(iv)) for US only*

Published:

— *with international search report*

— *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments*

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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: SILICONE VESICLES CONTAINING ACTIVES

(57) Abstract: A process is disclosed for preparing silicone visicle compositions containing a personal care or healthcare active, compositions prepared by the inventive process, and formulated products containing the silicone vesicles.

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INTERNATIONAL SEARCH REPORT

PCT/US2005/013326

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A61K7/00 A61K7/02 A61K7/48 A61K7/42 A61K9/127

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 780 113 A (L'OREAL) 25 June 1997 (1997-06-25) page 5, lines 15-39 page 2, lines 3-5,28-30,35-37 page 3, lines 40,41 page 4, lines 17,18,35-41	1,2,4-14
X	US 4 886 068 A (KANEKO ET AL) 12 December 1989 (1989-12-12) example 5	1,2
X	US 5 919 487 A (SIMONNET ET AL) 6 July 1999 (1999-07-06) column 2, lines 5-65; examples 1-5	1,2,4-14

-/--

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
11 August 2005	02. 11. 2005

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Sala-Jung, N

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2005/013326

Box II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☒ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

1, 2, 4-11, 12-14 (partly)

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☐ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. claims: 1,2,4-11,12-14 (partly)

Vesicle formation with an organopolysiloxane having a formula as defined in claim 2

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2. claims: 1,3,12-14 (partly)

Vesicle formation with an organopolysiloxane having a formula as defined in claim 3

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 120 778 A (SIMONNET ET AL) 19 September 2000 (2000-09-19) column 2, line 30 - column 3, line 33; examples 1-3	1,2,4-14
P,X	WO 2004/050045 A (DOW CORNING CORPORATION; HILL, RANDAL; LIN, ZUCHEN) 17 June 2004 (2004-06-17) example 9	1,2,4,7, 12-14

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0780113 A	25-06-1997	DE 69623423 D1	10-10-2002
		DE 69623423 T2	09-01-2003
		ES 2182958 T3	16-03-2003
		FR 2742674 A1	27-06-1997
		JP 3137592 B2	26-02-2001
		JP 9175930 A	08-07-1997
		US 5958433 A	28-09-1999
US 4886068 A	12-12-1989	NONE	
US 5919487 A	06-07-1999	DE 69600758 D1	12-11-1998
		DE 69600758 T2	04-03-1999
		EP 0780115 A1	25-06-1997
		ES 2125703 T3	01-03-1999
		FR 2742677 A1	27-06-1997
		JP 3001821 B2	24-01-2000
		JP 9175931 A	08-07-1997
US 6120778 A	19-09-2000	AT 229792 T	15-01-2003
		BR 9604724 A	01-09-1998
		CN 1156586 A	13-08-1997
		DE 69625452 D1	30-01-2003
		DE 69625452 T2	12-02-2004
		EP 0780114 A1	25-06-1997
		ES 2188734 T3	01-07-2003
		FR 2742676 A1	27-06-1997
		JP 3040355 B2	15-05-2000
		JP 9175933 A	08-07-1997
		PL 317635 A1	23-06-1997
WO 2004050045 A	17-06-2004	AU 2003293350 A1	23-06-2004
		EP 1567116 A1	31-08-2005